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Hi-G Lithium Thionyl Chloride Flat Cells for Artillery/Air Delivered Expendables

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Further development of the single-anode design has resulted in significant progress toward meeting the Hi-G performance goals. Capacities up to 14 ampere-hours have been demonstrated. Initial development of the double-anode design was begun and capacities up to 13 ampere-hours have been demonstrated.

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TABLE OF CONTENTS

FIGURES

I. Introduction

The objective of this program is the development of a lithium thionyl chloride flat cell for the titled application. The cell shall have a capacity of 14.6 ampere hours, with 66% delivered at 1.6 amps, after being subjected to severe conditions of shock (17,000 g's) and spin (16,000 rpm).

During the first quarter, exploratory tests were performed on the Altus AL-125, a cell which has a smaller diameter (1.25 inches) than the proposed Hi-G but has similar thickness and internal configuration. These tests led to the design of the first Hi-G prototype cells. The test results from these prototypes were presented in the first quarterly report.

During this quarter, the second quarter of the program, several more lots of prototype cells were constructed and tested. The cells in these subsequent lots were of both single-anode and double-anode designs, still using the 4.5 inch hardware described in the first report. They were tested under various load conditions, including: discharge entirely at the high (1.6 amp) rate, discharge entirely at the low (100 milliamp) rate and discharge switching from the high to the low rate.

Also completed during this quarter was a matrix of tests to investigate the relative and synergistic effects of several design parameters in the AL-125 (1.25 inch diameter) cell. These tests have served to define the most significant of these parameters and to indicate the appropriate trends for optimizing the Hi-G cell for its application.

II. Prototype Development: Single-Anode Configuration

Several lots of cells were built and tested during the period of this report which were based on the single-anode design. The cell cases were constructed from existing Altus AL-450 flat-cell hardware which has a 4.5 inch outside diameter and an external

height of 0.30 inches. For Hi-G prototype use, a flat circular shim of stainless steel, type 304, was welded inside the cell case in order to reduce the internal height to 0.150 inches, which is consistent with the proposed Hi-G dimensions. The anode and cathode diameters, 3.75 inches and 3.80 inches respectively, were sized similarly to conform to the Hi-G requirements. The anode thickness was fixed at 0.050 inches for all single-anode designs. The several single-anode lots built during this quarter differed only in the specifications of the cathodes used.

In the first lot built during this quarter (lot 101-10), the cathodes, of which there are two, one on either side of the single anode, weighed 4.0 grams each for a total cathode weight of 8.0 grams. The five cells in the lot were discharged at high and low rates as shown in Table 1.

TABLE 1

Cell No.	Load (Ohms)	Capacity (AH) to 2.5 Volts
101-10-1	2.04	10.7
-2	2.07	11.3
-3	34.00	12.9
-4	34.20	12.8
-5	34.00	14.3

The voltage profiles for cells 101-10-3 and 101-10-5 during discharge are shown in Figures 1 and 2 respectively. The ampere-hour capacities of these cells were close to that required for the Hi-G application and they indicated that the single-anode design could provide that capacity.

The effort to optimize the performance of the single-anode design was focused on the design of the cathode. This electrode is difficult to characterize because its performance is sensitive to many variables of which the most important are: the amount of material in each cathode, the porosity of the cathode, and the compressive force applied to the cathode during the assembly of the cell. In the groups

of prototype cells which followed lot 101-10, the weight and porosity of the cathodes were varied in order to realize an optimized configuration.

In lot 101-12, three cells were built wherein the total cathode weight per cell was six (6) grams. The cells were discharged at the high (approximately 1.5 ampere) rate and the results are shown in Table 2.

Table 2

Cell No.	Load (Ohms)	Capacity (AH) to 2.5 Volts
101-12-6	2.07	9.6
-7	2.04	9.4
-8	2.07	9.6

As might have been predicted from past experience, the capacities of these cells were not improved over those in lot 101-10. The low cathode densities are more suitable for cells optimized for low temperature and low discharge rate applications. The optimum cathode weight for the Hi-G application was thus shown to lie between six (6) and eight (8) grams per cell.

Lot 102-19 was built to test the capacities of cells wherein the total cathode weight per cell was seven (7) grams. These cells were tested with a two-step discharge: seven (7) hours at the two (2) ohm load with the remainder at the thirty-five (35) ohm load. The results of these tests appear in Table 3.

Table 3

Cell No.	2 Ohm Load Capacity	Subsequent 35 Ohm Capacity	Total Capacity
102-19-03	11.0 AH	1.8 AH	12.8 AH
-04	11.0	1.9	12.9
-05	10.5	1.4	11.9

These cells demonstrated some slight capacity improvement over lot 101-10 as would have been predicted from previous cathode optimization work.

In the last lot built during the period of this report, a somewhat new approach was tried, with encouraging results. The cells were built with cathode weights of six (6) grams per cell as in Lot 101-12 but they were subjected to an entirely different heating cycle in order to condition the cyanoacrylate antipassivation film applied to the anodes. These cells, too, were tested with a two-step discharge, the results of which appear in Table 4.

Table 4

Cell No.	2 Ohm Load Capacity	Subsequent 35 Ohm Capacity	Total Capacity
102-20-03	11.0 AH	2.4 AH	13.4 AH
-04	12.3	2.7	14.0

The voltage profile for cell 102-20-04 during discharge is shown in Figure 3. These cells performed significantly better than those in lot 101-12 and slightly better than those in lot 102-19. The reasons for this improvement are not fully understood at present. With these results, we are now confident of meeting Hi-G performance goals in the next quarter.

III. Prototype Development: Double-Anode Configuration

As an alternative to the single-anode design, the double-anode design has two advantages and two disadvantages. The first advantage is the reduction, by a factor of two (2), of the effective operating current densities witnessed by the electrodes. This reduction would benefit the utilization of the cathodes during discharge at the higher of the two Hi-G loads and would give the cell a capability to sustain somewhat higher discharge rates as well. The second advantage of the double-anode design is the possibility for slightly greater resistance to the Hi-G shock and spin environments due to the damping effect of the increased number of layers and to the reduced mass of each single component. The first disadvantage of the double-anode design is that the increase in the number of inert separator layers leaves less volume available for the cathodes. The second disadvantage is that the double-anode cell is significantly more difficult and more expensive to build.

In order to address the tradeoffs mentioned above, two different lots of cells were constructed: lot 102-21 with total cathode weights of 4.8 grams per cell and lot 103-17 with total cathode weights of 3.0 grams. The cells were discharged under different load conditions for which the results are shown in Table 5.

Table 5

Cell No.	Load (Ohms)	Capacity (AH) to 2.5 V	2.0V	Notes
102-21-1	2	7.6	10.3	3.0 grams total
-2	2	10.4	11.4	cathode weight
-3	2 ohms for (4 hrs)	7.5	7.6	
-4	then 35 ohms	5.4	5.6	
-5	35	10.5	12.3	
103-17-1	2	9.7	10.5	4.8 grams total
-2	2	10.0	11.3	cathode weight
-4	2 ohms for (6hrs)	12.7	13.3	
-5	then 35 ohms	12.1	12.5	

The capacities of lot 102-21 are quite erratic and indicate that the total cathode weights per cell were far below the useable minimum; the big variations corresponding to different discharge rates are especially indicative of poor electrode conductivity. The capacities of the cells in lot 103-17 are somewhat more promising. They are comparable to the first groups of single-anode cells and we might expect some improvement with optimization. More work on the double-anode design will be done in the next program quarter.

IV. Test of AL-125 Cells

At the beginning of this program quarter, a matrix of tests was performed using the Altus AL-125-HR cell. This cell has an internal configuration which is quite similar to the Hi-G cell although its diameter is 1.25 inches compared to approximately four (4) inches for the Hi-G. The object of these tests and their subsequent statistical evaluation was to identify, clearly, the correct trends and ranges for several design parameters critical to the optimization of the Hi-G cell.

Two (2) different levels of three (3) design parameters were tested starting with the thickness of the anode. While the quantity of lithium required to achieve a given capacity at a given rate can be directly calculated, the increased anode thickness in this experiment served as a uniform shim to change the effective internal volume of the cell and to change the compression applied to the other components of the cell. The weights of the cathodes were varied in a range corresponding to the cathode densities used for the Hi-G prototypes. Finally, the molarity of the electrolyte was varied, trying 1.0 molar electrolyte in order to determine the effect of an increased excess of thionyl chloride. The cells were tested at two different rates in order to determine the rate-sensitivity of the observed effects of the parametric variations. The different cell groups and design parameter variations are summarized in Table 6.

Table 6

Key:

	Parameter	Level 1	Level 2
A	Anode Thickness	.060 in	.080 in
B	Cathode Weight	.250g	.300g
C	Electrolyte	1.0M	1.6M
D	Load Resistor	100 ohm	50 ohm

	Matrix Schedule					Performance Results 70 mA		Performance Results 35 mA	
				Lot #	Cell #	Mean	SD	Mean	SD
1.	A1	B1	C1	101-06	21-40	1.053	.106	.927	.083
2.	A1	B1	C2	101-05	21-40	.873	.105	.866	.064
3.	A1	B2	C1	101-07	21-40	1.195	.056	1.231	.138
4.	A1	B2	C2	101-04	21-40	1.181	.198	1.175	.109
5.	A2	B1	C1	101-06	1-20	.900	.122	.868	.085
6.	A2	B1	C2	101-05	1-20	.844	.127	.866	.080
7.	A2	B2	C1	101-07	1-20	1.026	.105	.972	.102
8.	A2	B2	C2	101-04	1-20	.974	.158	.935	.102

Groups 3 and 4 did significantly better than the others. The small differences in molarity effect on the different lots indicates the lack of rate sensitivity in the design. Scale-up of these results suggets that the capacity attainable at these rates in a Hi-G design is approximately 14 ampere-hours. The range of capacity in the lots is significant enough to verify the feasibility of a 16 ampere-hour Hi-G cell. This experiment was effective in narrowing our range of design parameters. Additionally it demonstrated our ability to attain high capacity goals in this design.

This data can be analyzed using a matrix evaluation method. If discharge rate is incorporated as a variable there are four parameters each of which is tested at two levels. The matrix evaluation establishes an effect for the level change in each of the parameters and also establishes synergistic effects among the parameters. Table 7 is a summary of the results of the analysis.

The effect is a measure of the direction and magnitude of the change caused by the level shift of the parameter. Identifying a minimum magnitude for a significant effect is open to much interpretation. One established method uses the relation

$$(\mathrm{MIN}) = ts\left(\frac{2}{MK}\right)^{\frac{1}{2}}$$

where t = Student's t value at the desired probability level for the number of degrees of freedom in the etimate of s 1.833 for 90% confidence with d.f. = 9

s = pooled standard deviation of a single response observation. Best guess = 0.150

m = 2^{P-1} (p = 4 parameters)

k = number of replicates of each trial (10)

Minimum effect = 0.043 at 90% confidence level.

This suggests effects greater than 0.043 should be considered as significant. A more common-sense approach leads to the conclusion that this limit should be set higher. This is based on the fact that

increased rate shows an improvement which is significant according to the criteria found above. It is unlikely capacity will improve with increased rate. Thus it is preferred to choose a minimum effect limit around 0.060.

The data analysis leads to two effects which are very significant.

(1) Increased anode thickness decreases performance by approximately 28% of the mean.

(2) Increased cathode weight improves performance by approximately 19% of the mean.

All of the other effects appear to be unimportant.

Mean 0.993 AH

Parameter	Effect	
A	-0.279	A = increased anode thickness
B	+0.187	B = increased cathode weight
C	-0.057	C = increased $LiAlCl_4$ molarity
D	+0.052	D = increased discharge rate
AB	-0.079	
AC	+0.021	
AD	0.000	
BC	+0.018	
BD	-0.010	
CD	-0.018	
ABC	-0.051	
ABD	+0.031	
ACD	+0.001	
BCD	+0.025	
ABCD	-0.015	

TABLE 7

Summary of Matrix Analysis

Summary and Future Work

The single-anode prototypes have been developed to yield capacities proportional to those of AL-125. Considerable progress has been made toward realizing the performance goals of the Hi-G cell using prototype hardware and a single-anode configuration and it is expected that these goals will be met early in the next quarter. In addition, some work has been done on a double-anode prototype which will be further developed in the next quarter.

Hardware development for the Hi-G cell has been delayed. A first generation of hardware will be acquired and tested during the next quarter. Arrangements for shock testing have begun on prototype and first generation hardware and active cells. These tests will probably continue into the fourth quarter of the program.

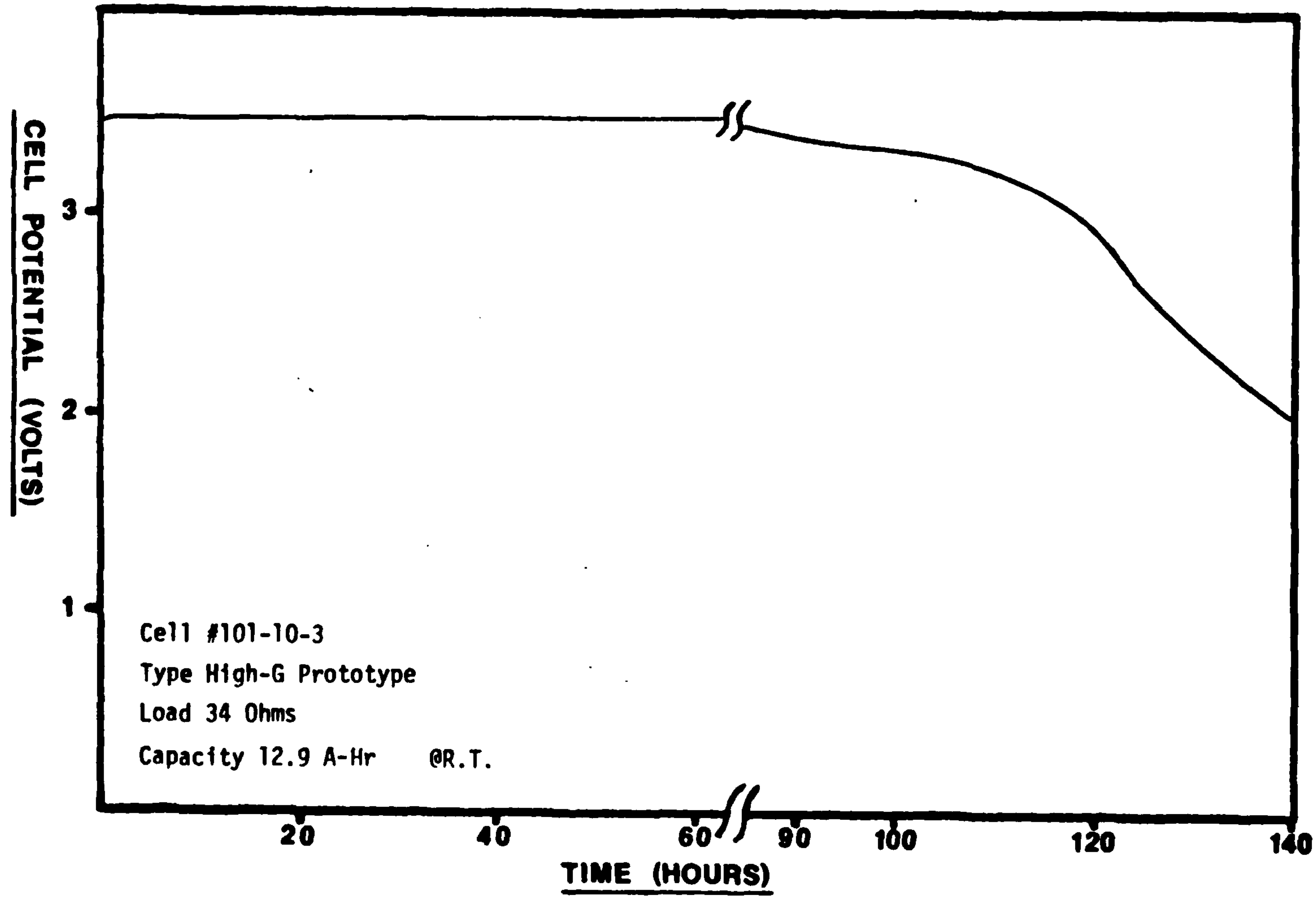

FIG.1 : Discharge Characteristics of the HIGH-G Prototype

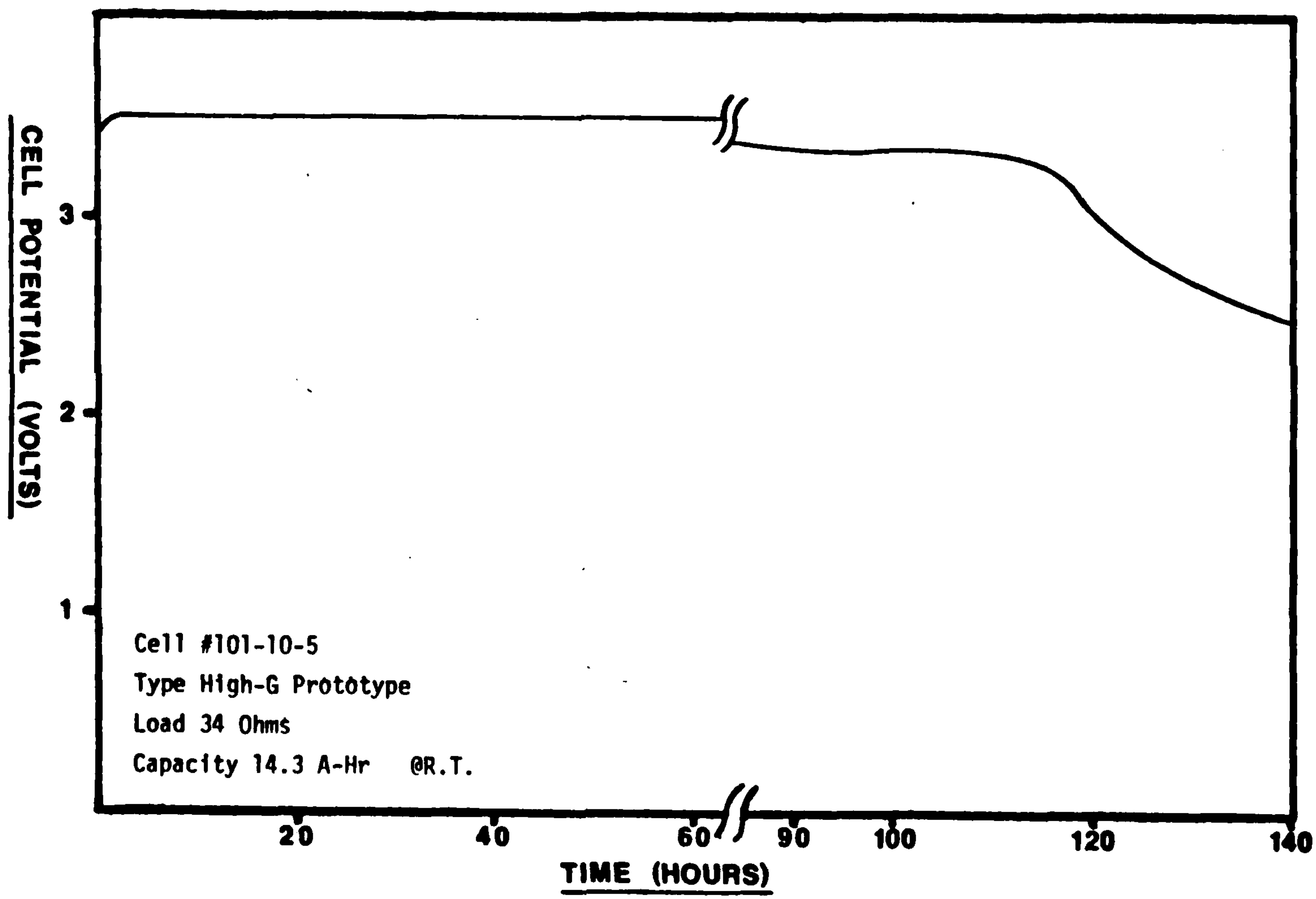

FIG.2 : **Discharge Characteristics of the HIGH - G Prototype**

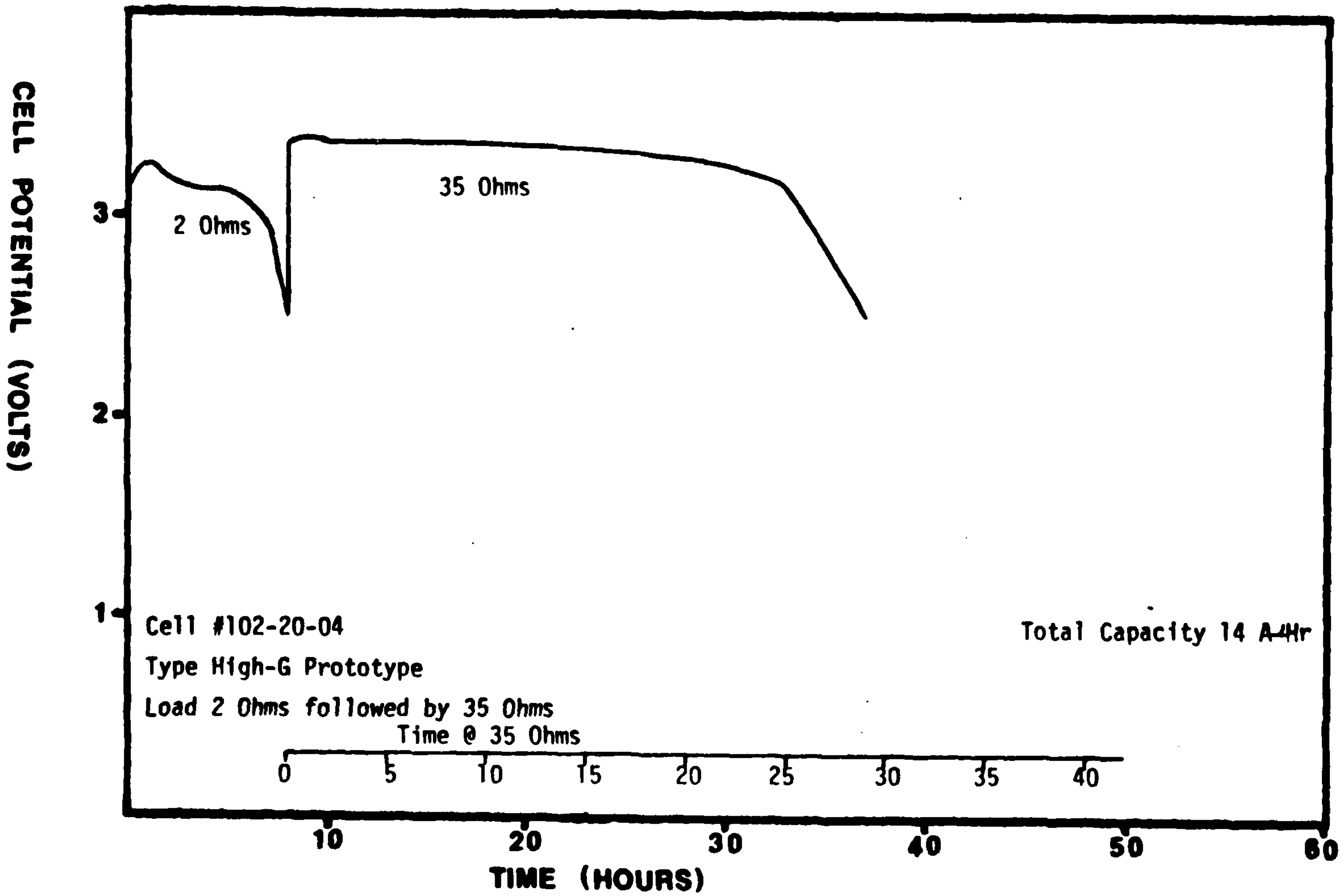

FIG. 3: **Mixed Rate Discharge Characteristics HIGH - G Prototype**